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Standard Practice for Microetching Metals and Alloys¹

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This standard has been approved for use by agencies of the U.S. Department of Defense.

^{ε1} NOTE—Originally approved date was editorially corrected to 1970 in footnote 1 in January 2016.

1. Scope

1.1 This practice covers chemical solutions and procedures to be used in etching metals and alloys for microscopic examination. Safety precautions and miscellaneous information are also included.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific cautionary statements, see 6.1 and Table 2.

2. Referenced Documents

2.1 *ASTM Standards:*²

D1193 Specification for Reagent Water

E7 Terminology Relating to Metallography

E2014 Guide on Metallographic Laboratory Safety

3. Terminology

3.1 *Definitions:*

3.1.1 For definition of terms used in this standard, see Terminology E7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *tint etch*—an immersion etchant that produces color contrast, often selective to a particular constituent in the microstructure, due to a thin oxide, sulfide, molybdate, chromate or elemental selenium film on the polished surface that reveals the structure due to variations in light interference effects as a function of the film thickness (also called a "stain etch").

¹ This practice is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.01 on Specimen Preparation.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2.2 *vapor-deposition interference layer method*—a technique for producing enhanced contrast between microstructural constituents, usually in color, by thin films formed by vacuum deposition of a dielectric compound (such as ZnTe, ZnSe, TiO₂, ZnS or ZnO) with a known index of refraction, generally due to light interference effects (also known as the "Pepperhoff method").

4. Summary of Practice

4.1 Table 1 is an alphabetical listing of the metals (including rare earths) and their alloys for which etching information is available. For each metal and alloy, one or more etchant numbers and their corresponding use is indicated. Alloys are listed as a group or series when one or more etchants are common to the group or series. Specific alloys are listed only when necessary. When more than one etchant number is given for a particular use, they are usually given in order of preference. The numbers of electrolytic etchants are *italicized* to differentiate them from nonelectrolytic etchants.

4.2 Table 2 is a numerical listing of all the etchants referenced in Table 1 and includes the composition and general procedure to be followed for each etchant.

4.3 To use the tables, look up the metal or alloy of interest in Table 1 and note the etchant numbers corresponding to the results desired. The etchant composition and procedure is then located in Table 2 corresponding to the etchant number.

4.4 If the common name of an etchant is known (Marble's, Vilella's, etc.), and it is desired to know the composition, Table 3 contains an alphabetical listing of etchant names, each coded with a number corresponding to the etchant composition given in Table 2.

5. Significance and Use

5.1 This practice lists recommended methods and solutions for the etching of specimens for metallographic examination. Solutions are listed to highlight phases present in most major alloy systems.

6. Safety Precautions

6.1 Before using or mixing any chemicals, all product labels and pertinent Material Safety Data Sheets (MSDS) should be

read and understood concerning all of the hazards and safety precautions to be observed. Users should be aware of the type of hazards involved in the use of all chemicals used, including those hazards that are immediate, long-term, visible, invisible, and with or without odors. See Guide E2014 on Metallographic Laboratory Safety for additional information on; Chemical Safety, Electrolytic Polishing/Etching and Laboratory Ventilation/Fume Hoods.

6.1.1 Consult the product labels and MSDSs for recommendations concerning proper protective clothing.

6.1.2 All chemicals are potentially dangerous. All persons using any etchants should be thoroughly familiar with all of the chemicals involved and the proper procedure for handling, mixing, and disposing of each chemical, as well as any combinations of those chemicals. This includes being familiar with the federal, state, and local regulations governing the handling, storage, and disposal of these chemical etchants.

6.2 Some basic suggestions for the handling and disposing of etchants and their ingredients are as follows:

6.2.1 When pouring, mixing, or etching, always use the proper protective equipment, (glasses, gloves, apron, etc.) and it is strongly recommended to always work under a certified and tested fume hood. This is imperative with etchants that give off noxious odors or toxic vapors that may accumulate or become explosive. In particular, note that solutions containing perchloric acid must be used in an exclusive hood equipped with a wash down feature to avoid accumulation of explosive perchlorates. See Guide E2014 on Metallographic Laboratory Safety for additional information on safety precautions for electrolytes containing perchloric acid..

6.2.2 No single type of glove will protect against all possible hazards. Therefore, a glove must be carefully selected and used to ensure that it will provide the needed protection for the specific etchant being used. In some instances it may be necessary to wear more than one pair of gloves to provide proper protection. Information describing the appropriate glove may be obtained by consulting the MSDS for the chemical being used. If that does not provide enough detailed information, contact the chemical manufacturer directly. Additionally, one can contact the glove manufacturer or, if available, consult the manufacturers glove chart. If the chemical is not listed or if chemical mixtures are being used, contact the glove manufacturer for a recommendation.

6.2.3 Use proper devices (glass or plastic) for weighing, mixing, containing, and storage of solutions. A number of etchants generate fumes or vapors and should only be stored in properly vented containers. Storage of fuming etchants in sealed or non-vented containers may create an explosion hazard.

6.2.4 When mixing etchants, always add reagents to the solvent unless specific instructions indicate otherwise.

6.2.5 When etching, always avoid direct physical contact with the etchant and specimen; use devices such as tongs to hold the specimen (and tufts of cotton, if used).

6.2.6 Methanol is a cumulative poison hazard. Where ethanol or methanol, or both are listed as alternates, ethanol is the preferred solvent. Methanol should be used in a properly designed chemical fume hood.

6.2.7 When working with HF always be sure to wear the appropriate gloves, eye protection and apron. Buying HF at the lowest useable concentration will significantly reduce risk. Additionally, it is recommended that a calcium gluconate cream or other appropriate HF neutralizing agent be available for use if direct skin contact of the etchant occurs.

6.2.8 The EPA states that human studies have clearly established that inhaled chromium (VI) is a human carcinogen, resulting in an increased risk of lung cancer. Animal studies have shown chromium (VI) to cause lung tumors via inhalation exposure. Therefore, when working with Cr(VI) compounds such as $K_2Cr_2O_7$ and CrO_3 always use a certified and tested fume hood. Additional information can be obtained at the EPA website³.

6.2.9 For safety in transportation, picric acid is distributed by the manufacturer wet with greater than 30% water. Care must be taken to keep it moist because dry picric acid is shock sensitive and highly explosive especially when it is combined with metals such as copper, lead, zinc, and iron. It will also react with alkaline materials including plaster and concrete to form explosive compounds. It should be purchased in small quantities suitable for use in six to twelve months and checked periodically for lack of hydration. Distilled water may be added to maintain hydration. It must only be stored in plastic or glass bottles with nonmetallic lids. If dried particles are noted on or near the lid, submerge the bottle in water to re-hydrate them before opening. It is recommended that any bottle of picric acid that appears dry or is of unknown vintage not be opened and that proper emergency personnel be notified.

6.2.10 Wipe up or flush any and all spills, no matter how minute in nature.

6.2.11 Properly dispose of all solutions that are not identified by composition and concentration.

6.2.12 Store, handle and dispose of chemicals according to the manufacturer's recommendations. Observe printed cautions on reagent bottles.

6.2.13 Information pertaining to the toxicity, hazards, and working precautions of the chemicals, solvents, acids, bases, etc. being used (such as material safety data sheets, MSDS) should be available for rapid consultation. A selection of useful books on this subject is given in Refs. (1-11)⁴.

6.2.14 Facilities which routinely use chemical etchants should have an employee safety training program to insure the employees have the knowledge to properly handle chemical etchants.

6.2.15 When working with etchants always know where the nearest safety shower, eye-wash station, and emergency telephone are located.

7. Miscellaneous Information

7.1 If you know the trade name of an alloy and need to know the composition to facilitate the use of Table 1, refer to a compilation such as Ref (12).

7.2 Reagent grade chemicals shall be used for all etchants. Unless otherwise indicated, it is intended that all reagents

³ <http://www.epa.gov/ttn/atw/hlthef/chromium.html>

⁴ The **boldface** numbers in parentheses refer to the list of references at the end of this standard.

conform to specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades, such as United States Pharmacopeia (USP), may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without detrimental effect.

7.2.1 Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of specification **D1193**. Experience has shown that the quality of tap water varies significantly and can adversely affect some etchants.

7.3 Methanol is usually available only as absolute methanol. When using this alcohol it is imperative that approximately 5 volume % of water is added whenever an etchant composition calls for 95 % methanol. Some of these etchants will not work at all if water is not present.

7.4 For conversion of small liquid measurements, there are approximately 20 drops/mL.

7.5 Etching should be carried out on a freshly polished specimen.

7.6 Gentle agitation of the specimen or solution during immersion etching will result in a more uniform etch.

7.7 The etching times given are only suggested starting ranges and not absolute limits.

7.8 In electrolytic etching, d-c current is implied unless indicated otherwise.

7.9 A good economical source of d-c current for small scale electrolytic etching is the standard 6-V lantern battery.

7.10 In electrolytic etching, the specimen is the anode unless indicated otherwise.

7.11 Do not overlook the possibility of multiple etching with more than one solution in order to fully develop the structure of the specimen.

7.12 Microscope objectives can be ruined by exposure to hydrofluoric acid fumes from etchant residue inadvertently left on the specimen. This problem is very common when the specimen or mounting media contain porosity and when the mounting material (such as Bakelite) does not bond tightly to the specimen resulting in seepage along the edges of the specimen. In all cases, extreme care should be taken to remove all traces of the etchant by thorough washing and complete drying of the specimen before placing it on the microscope stage.

7.13 Tint etchants (**13**, **14-16**) are always used by immersion, never by swabbing, as this would inhibit film formation. An extremely high quality polish is required as tint etchants will reveal remaining polishing damage even if it is not visible with bright field illumination. After polishing, the surface must be carefully cleaned. Use a polyethylene beaker to contain the etchant if it contains fluorine ions (for example, etchants containing ammonium bifluoride, NH_4FHF). The specimen is placed in the solution using tongs, polished face up. Gently agitate the solution while observing the polished surface. After coloration begins, allow the solution to settle and remain motionless. Remove the specimen from the etchant when the surface is colored violet, rinse and dry. A light pre-etch with a general-purpose chemical etchant may lead to sharper delineation of the structure after tint etching.

7.14 Specimens should be carefully cleaned before use of a vapor-deposition interference film (“Pepperhoff”) method (**13**, **14-17**). A light pre-etch, or a slight amount of polishing relief, may lead to sharper delineation of the constituents after vapor deposition. The deposition is conducted inside a vacuum evaporator of the type used to prepare replicas for electron microscopy. One or several small lumps of a suitable dielectric compound with the desired index of refraction is heated under a vacuum until it evaporates. A vacuum level of 1.3 to 0.013 Pa (10^{-3} to 10^{-5} mm Hg) is adequate and the polished surface should be about 10–15 cm beneath the device that holds the dielectric compound. Slowly evaporate the lumps and observe the surface of the specimen. It may be helpful to place the specimen on a small piece of white paper. As the film thickness increases, the surface (and the paper) will become colored with the color sequence changing in the order yellow, green, red, purple, violet, blue, silvery blue. Stop the evaporation when the color is purple to violet, although in some cases, thinner films with green or red colors have produced good results.

7.15 Metals Handbook (**18**) provides additional advice on etching solutions and techniques for various alloys.

8. Precision and Bias

8.1 It is not possible to specify the precision or bias of this practice since quantitative measurements are not made.

9. Keywords

9.1 etch; etchant; interference method; metallography; metals; microetch; microscope; microstructure; Pepperhoff method; tint etch



TABLE 1 Etchants for Metals

NOTE 1—It is strongly recommended to always mix and use etchants under a certified and tested fume hood.

NOTE 2—Electrolytic etchants are *italicized*.

Metal	Etchants	Uses
<i>Aluminum Base:</i>		
Pure Al	1a, 2, 3 4, 5 1b	general structure grain structure under polarized light grain boundaries and slip lines
1000 series	1a, 3, 2 4, 5 6, 7	general structure grain structure under polarized light phase identifications
2000 series	3, 2, 1a 8a, 6, 7	general structure phase identifications
3000 series	3, 1a 4, 5 8a, 6, 7	general structure grain structure under polarized light phase identifications
4000 series	3, 1a	general structure
5000 series	3, 1a, 2, 6, 8a 4, 5	general structure grain structure under polarized light
6000 series	3, 1a, 2, 6, 8a, 222 4, 5 1a, 2, 7, 6, 8a	general structure grain structure under polarized light phase identifications
7000 series	3, 1a, 2 4, 5 3b, 6	general structure grain structure under polarized light phase identifications
<i>Beryllium Base:</i>		
Pure Be	9, 10	general structure via polarized light
Be alloys	11	general structure
<i>Chromium Base:</i>	12, 13c	general structure
<i>Cobalt Base:</i>		
Pure Co	14, 15, 16, 17	general structure
Hard-facing and tool metals	18, 19, 20	general structure
High-temperature alloys	20, 18, 16, 21, 22b, 24, 25 19	general structure phase identification
<i>Columbium Base</i> (see niobium base)		
<i>Copper Base:</i>		
Pure Cu	26, 27, 28, 29, 30, 31d, 32, 33, 34b, 35, 36, 37, 38, 39, 40, 41, 42, 8b, 210, 215 43, 28	general structure chemical polish and etch
Cu-Al (aluminum bronze)	44, 31d, 34b, 35, 36, 37, 38, 39, 40, 45, 215	general structure
Cu-Be	46, 41, 45	general structure
Cu-Cr	41	general structure
Cu-Mn	41	general structure
Cu-Ni	34, 47, 48, 40, 49, 50	general structure
Cu-Si	41	general structure
Cu-Sn (tin bronze)	51, 52	general structure
Admiralty metal	8b	general structure
Gilding metal		
Cartridge brass		
Free-cutting brass		
Nickel silver	31d, 32, 33, 41, 42, 49	general structure
Cu alloys	26, 27, 28, 29, 30, 44, 41, 31d, 32, 33, 34b, 35, 36, 37, 38, 39, 210, 215 53, 43, 28, 49 42, 49, 210 54	general structure chemical polish and etch darkens beta in alpha-beta brass etching of cold worked brass
<i>Dysprosium Base:</i>	55, 56	general structure

TABLE 1 *Continued*

Metal	Etchants	Uses
<i>Erbium Base:</i>	55, 56	general structure
<i>Gadolinium Base:</i>	55, 56, 57	general structure
<i>Germanium Base:</i>	58, 59, 60	general structure
<i>Gold Base:</i>		
Pure Au	61, 62	general structure
	63	chemical polish and etch
Au alloys	64b, 62	general structure
	63	chemical polish and etch
>90 % noble metals	61	general structure
<90 % noble metals	65	general structure
<i>Hafnium base:</i>	66, 67, 68, 69, 70	general structure
	71	grain structure under polarized light
	72	chemical polish and etch
<i>Holmium Base:</i>	55, 56	general structure
<i>Iridium Base:</i>	73c	general structure
<i>Iron Base:</i>		
Pure Fe	74a	grain boundaries
	75	substructure
	210	colors ferrite grains
Fe + C	76, 74a, 77, 78, 79	general structure
and	74a, 77, 31a, 223	ferrite grain boundaries
Fe + <1C + <4 % additions	80, 81, 82	prior austenitic grain boundaries in martensitic and bainitic steels
	78, 222a	untempered martensite
	31b, 78	carbides and phosphides (matrix darkened, carbides and phosphides remain bright)
	83	cementite attacked rapidly, sustenite less, ferrite and iron phosphide least
	84	overheating and burning
	85	stains carbides
	86	chemical polish-etch
	210, 211	colors ferrite
	213, 214	colors carbides
	216	colors lath martensite in low-carbon high-alloy grades
	222b	for dual phase steels; reveals pearlite, darkens martensite and outlines austenite
Fe + 4–12 Cr	80, 87, 88, 89, 90, 91, 79, 210	general structure
	86	chemical polish-etch
Fe + 12–30 Cr + <6 Ni (400 Series)	80, 87, 88, 89, 34, 40, 92, 93, 94, 95, 91, 226	general structure
	96, 97, 98	signs phase
	31c	carbides
	86	chemical polish-etch
	219	grain boundary etch
	220	darkens delta ferrite
Fe + 12–20 Cr + 4–10 Ni + <7 % other elements (controlled transformation, precipitation hardening, stainless maraging alloys)	80, 31c, 89, 99, 100, 91	general structure
	31c	carbides
	86	chemical polish-etch
	220	darkens delta ferrite
Fe + 15–30 Cr + 6–40 Ni + <5 % other elements (300 Series)	13b, 89, 87, 88, 83a, 80, 94, 95, 91, 101, 212, 221, 226	general structure
	13a, 102, 31c, 48c, 213	carbides and sensitization
and	48, 96, 97, 98	stains sigma phase
Fe + 16–25 Cr + 3–6 Ni + 5–10 Mn (200 series)	103, 104, 98	delineates sigma phase and welds of dissimilar metals
	103, 104	chemical polish-etch
	86	grain boundary etch (no twins)
	219	darkens delta ferrite
	220	
High temperature	89, 25, 105, 106, 97, 212, 221	general structure
	107, 108, 213	γ' precipitate
	86	chemical polish-etch
Nonstainless maraging steels	109, 89, 99, 100, 221	general structure
	83b	grain boundaries
	86	chemical polish-etch



TABLE 1 Continued

Metal	Etchants	Uses
Tool steels	74a, 80, 14 110 210, 211 214, 214 224, 225	general structure grain boundaries in tempered tool steel colors ferrite, lower alloy grades colors cementite carbides attacked and colored
Superalloys	86, 87, 94, 221, 226 111 111	general etch general structure γ' depletion
<i>Lead Base:</i> Pure Pb	57, 112 113	general structure for alternate polishing and etching
Pb + <2 Sb	114, 115, 57, 74b 113	general structure for alternate polishing and etching
Pb + >2 Sb	114, 57, 74b 113	general structure for alternate polishing and etching
Pb + Ca	112 113	general structure for alternate polishing and etching
Pb alloys Babbitt	116, 117b 74b	general structure general structure
<i>Magnesium Base:</i> Pure Mg	118, 119, 74a, 120, 121, 122 123	general structure stain-free polish-etch
Mg-Mn	119, 74a, 124, 122	general structure
Mg-Al, Mg-Al-Zn (Al + Zn <5 %)	118, 119, 74a, 125, 124, 123, 122 120, 125, 126, 127 124, 126, 127	general structure general structure phase identification grain structure
Mg-Al, Mg-Al-Zn (Al + Zn >5 %)	118, 119, 74a, 125, 124, 121, 122 120, 125, 126, 127	general structure phase identification general structure
Mg-Zn-Zr and Mg-Zn-Th-Zr	118, 119, 74a, 1d, 128, 124, 126, 127, 121, 122 120, 121	phase identification general structure
Mg-Th-Zr and Mg-Rare Earth-Zr	118, 119, 74a, 1d, 124, 127, 121, 122 120, 121	phase identification
<i>Molybdenum Base:</i> As cast	98c, 129, 130, 131 132a	general structure chemical polish prior to etching
<i>Nickel Base:</i> Pure Ni and high Ni alloys	133, 134, 47, 135, 136, 25, 108, 31c 137	general structure grain boundary sulfidation
Ni-Ag	38, 138, 50, 139	general structure
Ni-Al	50, 140, 141, 142, 89, 143	general structure
Ni-Cr	144, 50, 83, 134, 145, 98, 146, 147, 13a	general structure
Ni-Cu	38, 138, 50, 133, 140, 25, 134, 47, 48b, 94, 108, 34	general structure
Ni-Fe	50, 140, 141, 83, 134, 148, 40, 107, 149 74e, 25, 150	general structure orientation pitting
Ni-Mn	74e	general structure
Ni-Mo	143	general structure
Ni-Ti	143, 151, 50, 133	general structure
Ni-Zn	152	general structure
Superalloys	94, 105, 138, 153, 12, 87, 89, 212, 226 25, 94 107, 111, 13a 133 154 19b, 155, 156 22a 157 107 154 18 213	general structure grain size reveals microstructural inhomogeneity grain boundary sulfidation fine precipitation structure differential matrix and nonmetallic staining for passive alloys (for example, UNS Alloy N06625) specific for UNS Alloy N10004 submicroscopic structure in aged super-alloys particu- larly for electron microscopy. Stains the matrix when γ' precipitates are present γ' banding pre-etch activation for passive specimens colors carbide and γ'



TABLE 1 Continued

Metal	Etchants	Uses
<i>Niobium (Columbium) Base:</i>	129, 66, 158, 159, 160, 161, 162, 163 164, 129, 160	general structure grain boundaries
<i>Osmium Base:</i>	165a 165a	general structure etch-polishing for viewing grains with polarized light
<i>Palladium Base:</i>		
Pure Pd	61, 166, 62, 165a	general structure
Pd alloys	166, 64a, 62, 165a	general structure
>90 % noble metals	61	general structure
<90 % noble metals	65	general structure
<i>Platinum Base:</i>		
Pure Pt	64a, 73a 167	general structure electrolytic polish and etch
Pt Alloys	64b, 73a 167	general structure electrolytic polish and etch
>90 % noble metals	61	general structure
<90 % noble metals	65	general structure
Pt-10 % Rh	168	general structure
<i>Plutonium Base:</i>	169	general structure
<i>Rhenium Base:</i>	13b, 98c, 132b, 170a	general structure
<i>Rhodium Base:</i>	171	general structure
<i>Ruthenium Base:</i>	73b 73b	general structure etch-polishing for viewing grains with polarized light
<i>Silver Base:</i>		
Pure Ag	172, 173, 62	general structure
Ag alloys	65, 61, 174, 175, 62	general structure
Ag-Cu alloys	130	general structure
Ag-Pd alloys	173	general structure
Ag solders	173, 176	general structure
<i>Tantalum Base:</i>		
Pure Ta	177	general structure
Ta alloys	159, 66, 178, 163, 161, 179 164 158	general structure grain boundaries and inclusions grain boundaries—retains carbide precipitate
<i>Thorium Base:</i>		
Pure Th	185	general structure
Th alloys	185	general structure
<i>Tin Base:</i>		
Pure Sn	74d, 180, 151 181	general structure grain boundaries
Sn-Cd	74d	general structure
Sn-Fe	74d, 177a	general structure
Sn-Pb	182, 183, 74b 116	general structure darkens Pb in Sn-Pb eutectic
Sn coatings (on steel)	183	general structure
Babbitts	184	general structure
Sn-Sb-Cu	74b	general structure
<i>Titanium Base:</i>		
Pure Ti	186, 187, 67, 68, 69, 217 188 72	general structure removes stain chemical polish and etch
Ti-5 Al-2,5 Sn	189	reveals hydrides
Ti-6 Al-6 V-2 Sn	190	Stains alpha and transformed beta, retained beta re mains white
Ti-Al-Zr	191	general structure
Ti-8Mn	192	general structure
Ti-13 V-11 Cr-3 Al (aged)	192	general structure
Ti-Si	193	general structure
Ti alloys	186, 187, 192, 194, 158, 132b, 1c, 67, 68, 69, 3a, 218 11, 1c 72, 192, 178 170a 188	general structure reveals alpha case chemical polish and etch outlines and darkens hydrides in some alloys removes stain
<i>Tungsten Base:</i>		

TABLE 1 *Continued*

Metal	Etchants	Uses
Pure W	98c, 131	general structure
As cast	132a	chemical polish prior to etching
W-Th	209	general structure
<i>Uranium Base:</i>		
Pure U	67, 69, 195, 196	general structure
U + Zr	68	general structure
U beryllides	170a	general structure
U alloys	67, 69, 195, 96	general structure
	207	carbides
<i>Vanadium Base:</i>		
Pure V	170b, 165b	general structure
	197, 198	grain boundaries
V alloys	199, 198	general structure
<i>Zinc Base:</i>		
Pure Zn	200a	general structure
Zn-Co	177	general structure
Zn-Cu	201	general structure
	203	distinguishes gamma (γ) and epsilon (ε)
Zn-Fe	74a	structure of galvanized sheet
Die castings	202	general structure
<i>Zirconium Base:</i>	66, 67, 204, 68, 69, 205	general structure
	206	electrolytic polish and etch
	71	grain structure under polarized light
	72	chemical polish and etch



TABLE 2 Numerical List of Etchants

NOTE 1—It is strongly recommended to always mix and use etchants under a certified and test fume hood.

Etchant	Composition	Procedure
1	1 mL HF 200 mL water	(a) Swab with cotton for 15 s. (b) Alternately immerse and polish several minutes. (c) Immerse 3–5 s. (d) Immerse 10–120 s.
2	3 mL HF 100 mL water	(a) Swab 10 s to reveal general structure. (b) Immerse 15 min, wash 10 min in water to form film with hatching which varies with grain orientation.
3	2 mL HF 3 mL HCl 5 mL HNO ₃ 190 mL water	(a) Immerse 10–20 s Wash in stream of warm water. Reveals general structure. (b) Dilute with 4 parts water-colors constituents—mix fresh.
4	24 mL H ₃ PO ₄ 50 mL Carbitol (diethylene glycol monoethyl ether) 4 g boric acid 2 g oxalic acid 10 mL HF 32 mL water	Electrolytic: Use carbon cathode raising d-c voltage from 0–30 V in 30 s. Total etching time 3 min with agitation. Wash and cool. Repeat if necessary.
5	5 g HBF ₄ 200 mL water	Electrolytic: Use Al, Pb, or stainless steel cathode. Anodize 1–3 min, 20–45 V d-c. At 30 V, etch for 1 min.
6	25 mL HNO ₃ 75 mL water	Immerse 40 s at 70°C (160°F). Rinse in cold water.
7	10–20 mL H ₂ SO ₄ 80 mL water	Immerse 30 s at 70°C (160°F). Rinse in cold water.
8	10 mL H ₃ PO ₄ 90 mL water	(a) Immerse 1–3 min at 50°C (120°F). (b) Electrolytic at 1–8 V for 5–10 s.
9	3–4 g sulfamic acid 5 drops HF 100 mL water	Use just prior to the last polishing operation. It is not intended as a final etchant. The specimen is examined as polished under polarized light.
10	10 mL HF 90 mL methanol (90 %)	Immerse 10–30 s.
11	2 mL HF 100 mL water	Immerse or swab few seconds to a minute.
12	20 mL HNO ₃ 60 mL HCl	Use a certified and tested hood. Do not store. Immerse or swab 5–60 s.
13	10 g oxalic acid 100 mL water	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen.
14	10 mL HNO ₃ 90 mL methanol (95 %)	Immerse few seconds to a minute.
15	15 mL HNO ₃ 15 mL acetic acid 60 mL HCl 15 mL water	Use a certified and tested hood. Age before use. Immerse 5–30 s. May be used electrolytically.
16	5–10 mL HCl 100 mL water	Electrolytic at 3 V for 2–10 s.
17	5 mL HCl 10 g FeCl ₃ 100 mL water	Electrolytic at 6 V for few seconds.
18	2–10 g CrO ₃ 100 mL water	Use a certified and tested hood. Electrolytic at 3 V for 2–10 s.



TABLE 2 Continued

Etchant	Composition	Procedure
19	A 8 g NaOH 100 mL water B Saturated aqueous solution of KMnO_4	Immerse in freshly mixed Solutions A + B (1:1) for 5–10 s. If surface activation is necessary, first use Etch #18, then rinse in water. While still wet, immerse in Solutions A + B (1:1). Mixture of solutions A + B has 15-min useful life. Note: KMnO_4 is an aggressive staining agent.
20	5 mL H_2O_2 (30 %) 100 mL HCl	Use a certified and tested hood. <i>Mix fresh.</i> Immerse polished face up for few seconds.
21	1 g CrO_3 140 mL HCl	Use a certified and tested hood. To mix, add the HCl to CrO_3 . Electrolytic at 3 V for 2–10 s.
22	100 mL HCl 0.5 mL H_2O_2 (30 %)	Use a certified and tested hood. Do not store. (a) Immerse or swab $\frac{1}{2}$ –3 min. Add H_2O_2 dropwise to maintain action. (b) Electrolytic, 4 V, 3–5 s.
23	5 mL HCl 95 mL ethanol (95 %) or methanol (95 %)	Electrolytic at 6 V for 10–20 s.
24	5 mL HNO_3 200 mL HCl 65 g FeCl_3	Use a certified and tested hood. Immerse few seconds.
25	10 g CuSO_4 50 mL HCl 50 mL water	Immerse or swab 5–60 s. Made more active by adding few drops of H_2SO_4 just before use.
26	5 g FeCl_3 10 mL HCl 50 mL glycerol 30 mL water	Swab 16–60 s. Activity may be decreased by substituting glycerol for water.
27	1 g KOH 20 mL H_2O_2 (3 %) 50 mL NH_4OH 30 mL water	Dissolve KOH in water, then slowly add NH_4OH to solution. Add 3 % H_2O_2 last. Use fresh—immerse few seconds to a minute.
28	1 g FeNO_3 100 mL water	Swab or immerse few seconds to a minute.
29	1 g $\text{K}_2\text{Cr}_2\text{O}_7$ 4 mL H_2SO_4 50 mL water	Use a certified and tested hood. Add 2 drops of HCl just before using. Swab few seconds to a minute.
30	25 mL NH_4OH 25 mL water 50 mL H_2O_2 (3 %)	Mix NH_4OH and water before adding H_2O_2 . Must be used fresh. Swab 5–45 s.
31	10 g ammonium persulfate 100 mL water	(a) Swab or immerse to 5 s. (b) Immerse to 2 min to darken matrix to reveal carbides and phosphides. (c) Electrolytic at 6 V for few seconds to a minute. (d) Immerse 3–60 s. Can be heated to increase activity.
32	60 g CrO_3 100 mL water	Use a certified and tested hood. Saturated solution. Immerse or swab 5–30 s.
33	10 g CrO_3 2–4 drops HCl 100 mL water	Use a certified and tested hood. Add HCl just before use. Immerse 3–30 s. Phases can be colored by Nos. 35, 36, 37.
34	5 g FeCl_3 50 mL HCl 100 mL water	(a) Immerse or swab few seconds to few minutes. Small additions of HNO_3 activate solution and minimize pitting. (b) Immerse or swab few seconds at a time. Repeat as necessary.
35	20 g FeCl_3 5 mL HCl 1 g CrO_3 100 mL water	Use a certified and tested hood. Immerse or swab few seconds at a time until desired results are obtained.
36	25 g FeCl_3 25 mL HCl 100 mL water	Immerse or swab few seconds at a time until desired results are obtained.



TABLE 2 Continued

Etchant	Composition	Procedure
37	1 g FeCl ₃ 10 mL HCl 100 mL water	Immerse or swab few seconds at a time until desired results are obtained
38	8 g FeCl ₃ 25 mL HCl 100 mL water	Swab 5–30 s.
39	5 g FeCl ₃ 10 mL HCl 1 g CuCl ₂ 0.1 g SnCl ₂ 100 mL water	Immerse or swab few seconds at a time until desired results are obtained.
40	5 g FeCl ₃ 16 mL HCl 60 mL ethanol (95 %) or methanol (95 %)	Immerse or swab few seconds to few minutes.
41	2 g K ₂ Cr ₂ O ₇ 8 mL H ₂ SO ₄ 4 drops HCl 100 mL water	Use a certified and tested hood. Add the HCl just before using. Immerse 3–60 s.
42	10 g cupric ammonium chloride 100 mL water NH ₄ OH	Add NH ₄ OH to solution until neutral or slightly alkaline. Immerse 5–60 s.
43	20 mL NH ₄ OH 1 g ammonium persulfate 60 mL water	Immerse 5–30 s.
44	50 mL NH ₄ OH 20–50 mL H ₂ O ₂ (3 %) 0–50 mL water	Use fresh. Peroxide content varies directly with copper content of alloy to be etched. Immerse or swab to 1 min. Film on etched aluminum bronze removed by No. 82.
45	1 g CrO ₃ 100 mL water	Use a certified and tested hood. Electrolytic at 6 V for 3–6 s. Use aluminum cathode.
46	15 mL NH ₄ OH 15 mL H ₂ O ₂ (3 %) 15 mL water 4 pellets NaOH	When mixing, add NaOH pellets last. For best results use before pellets have dissolved.
47	5 g NaCN or KCN 5 g (NH ₄) ₂ S ₂ O ₂ 100 mL water	Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact.
48	10 g NaCN 100 mL water	Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Electrolytic at 6 V: (a) 5 s for sigma. (b) 30 s for ferrite and general structure. (c) to 5 min for carbides.
49	3 g FeSO ₄ 0.4 g NaOH 10 mL H ₂ SO ₄ 190 mL water	Electrolytic at 8–10 V (0.1 A) for 5–15 s.
50	5 mL acetic acid 10 mL HNO ₃ 85 mL water	Use a certified and tested hood. Do not store. Electrolytic at 1.5 V for 20 to 60 s. Use platinum wires.
51	2 g FeCl ₃ 5 mL HCl 30 mL water 60 mL ethanol or methanol	Immerse few minutes.
52	1 g sodium dichromate 1 g NaCl 4 mL H ₂ SO ₄ 250 mL water	Swab few seconds.
53	1–5 mL NH ₄ OH 100 mL water	Immerse 5–60 s.



TABLE 2 Continued

Etchant	Composition	Procedure
54	1 g ammonium acetate 3 g sodium thiosulfate 7 mL NH ₄ OH 1300 mL water	Electrolytic at 0.3 A/cm ² for 5–30 s.
55	1 mL H ₂ SO ₄ 15 mL HNO ₃ 10 mL acetic acid 5 mL H ₃ PO ₄ 20 mL lactic acid	Use a certified and tested hood. Swab gently 10–15 s. Rinse with methanol and blow dry. Helps to chemically polish. If final etch is too mild, follow with No. 98. Do not store.
56	30 mL HNO ₃ 10 mL H ₃ PO ₄ 20 mL acetic acid 10 mL lactic acid	Use a certified and tested hood. Swab gently 5–15 s. Rinse with ethanol or methanol and blow dry. Do not store.
57	75 mL acetic acid 25 mL H ₂ O ₂ (30 %)	Use a certified and tested hood. Immerse 6–15 s. Do not store.
58	25 mL HF 25 mL HNO ₃ 5 mL water	Swab 3–20 s.
59	2 g AgNO ₃ 40 mL water 40 mL HF 20 mL HNO ₃	Mix AgNO ₃ and water, then add HF and HNO ₃ . Swab ½ –2 min.
60	25 mL HNO ₃ 15 mL acetic acid 15 mL HF 5–7 drops bromine	Use a certified and tested hood. Do not store. Let stand ½ h before using. Swab 3–20 s.
61	60 mL HCl 40 mL HNO ₃	Use a certified and tested hood. Immerse few seconds to a minute.
62	1–5 g CrO ₃ 100 mL HCl	Use a certified and tested hood. Vary composition of reagent and aging of reagent after mixing to suit alloy. Swab or immerse few seconds to a minute.
63	0.1 g CrO ₃ 10 mL HNO ₃ 100 mL HCl	Use a certified and tested hood. Swab few seconds to a minute.
64	5 mL HNO ₃ 25 mL HCl 30 mL water	(a) Immerse 1–5 min. (b) Use hot. Will form chloride film on gold alloys if much silver is present. Ammonia will remove film.
65	A 10 g ammonium persulfate 100 mL water B 10 g KCN 100 mL water	Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Mix 1 + 1 mixture of Solutions A and B just before use. (A mixture of 5 drops of each will cover the surface of a 1 in. dia. mount.) Immerse ½ – 2 min.
66	30 mL HF 15 mL HNO ₃ 30 mL HCl	Use a certified and tested hood. Swab 3–10 s or immerse to 2 min.
67	10 mL perchloric acid 10 mL 2-butoxyethanol 70 mL ethanol (95 %) 10 mL water	Use in wash down/perchloric rated fume hood. Precaution—Keep cool when mixing and use. Electrolytic at 30–65 V for 10–60 s.
68	3 mL perchloric acid 35 mL 2-butoxyethanol 60 mL methanol (absolute)	Use in wash down/perchloric rated fume hood. Precaution—Keep cool when mixing and use. Electrolytic at 60–150 V for 5–30 s.
69	5 mL perchloric acid 80 mL acetic acid	Use in wash down/perchloric rated fume hood. Precaution—Keep cool when mixing and use. Electrolytic at 20–60 V for 1–5 min. Do not store.
70	5 mL HF 2 mL AgNO ₃ (5 %) 200 mL water	Swab for 5–60 s.
71	5 mL HF 95 mL water	Add 5–10 drops of this solution on the final polishing wheel which has been charged with the polishing solution. The specimen is polished on this wheel until the surface turns black. Distilled water is then slowly added to the wheel and polishing continued until the surface is bright. At this time the specimen should be ready for examination via polarized light. Note—Use inert substance between cloth and wheel to prevent attack of the wheel. Wear appropriate gloves.



TABLE 2 Continued

Etchant	Composition	Procedure
72	10 mL HF 45 mL HNO ₃ 45 mL water	Swab for 5–20 s.
73	20 mL HCl 25 g NaCl 65 mL water	Electrolytic etch—use carbon cathode and platinum wire connection to specimen. (a) 6 V ac for 1 min. (b) 5 V–20 V ac for 1–2 min. (c) 20 V ac for 1–2 min. For etch-polishing, use shorter times. After etching, water rinse, alcohol rinse, and dry.
74	1–5 mL HNO ₃ 100 mL ethanol (95 %) or methanol (95 %)	Etching rate is increased, sensitivity decreased with increased percentage of HNO ₃ . (a) Immerse few seconds to a minute. (b) Immerse 5–40 s in 5 % HNO ₃ solution. To remove stain, immerse 25 s in 10 % HCl-methanol solution. (c) For Inconels and Nimonic, use 5 mL HNO ₃ solution—electrolytic at 5–10 V for 5–20 s. (d) Swab or immerse several minutes. (e) Swab 5–60 s. HNO ₃ may be increased to 30 mL in methanol only depending on alloy. (Ethanol is unstable with over 5 % HNO ₃ .) Do not store.
75	5 g picric acid 8 g CuCl ₂ 20 mL HCl 200 mL ethanol (95 %) or methanol (95 %)	Immerse 1–2 s at a time and immediately rinse with methanol. Repeat as often as necessary. (Long immersion times will result in copper deposition on surface.)
76	4 g picric acid 100 mL ethanol (95 %) or methanol (95 %)	Composition given will saturate with picric acid. Immerse few seconds to a minute or more. Adding a wetting agent such as zepherin chloride will increase response.
77	10 g picric acid 5 drops HCl 100 mL ethanol (95 %) or methanol (95 %)	Composition given will saturate the solution with picric acid. Immerse few seconds to a minute or more.
78	10 g potassium metabisulfite 100 mL water	Immerse 1–15 s. Better results are sometimes obtained by first etching lightly with No. 76 or 74.
79	40 mL HCl 5 g CuCl ₂ 30 mL water 25 mL ethanol (95 %) or methanol (95 %)	Swab few seconds to a minute.
80	5 mL HCl 1 g picric acid 100 mL ethanol (95 %) or methanol (95 %)	Immerse or swab few seconds to 15 min. Reaction may be accelerated by adding a few drops of 3 % H ₂ O ₂ . Optional (for prior austenite grain boundaries)—temper specimen at 600–900°F prior to preparation.
81	2 g picric acid 1 g sodium tridecylbenzene sulfonate. 100 mL water	Composition given will saturate the solution with picric acid. (a) Immerse few seconds to a minute. (b) Immerse to 15 min with occasional swabbing for heavy grain boundary attack.
82	5 g FeCl ₃ 5 drops HCl 100 mL water	Immerse 5–10 s.
83	10 g CrO ₃ 100 mL water	Use a certified and tested hood—(a) Electrolytic at 6 V for 5–60 s. Attacks carbides. (b) Electrolytic at 6 V for 3–5 s.
84	10 mL H ₂ SO ₄ 10 mL HNO ₃ 80 mL water	Use a certified and tested hood. Precaution—Add H ₂ SO ₄ slowly to water and cool, then add HNO ₃ . Immerse 30 s. Swab in running water. Repeat three times and repolish lightly.
85	2 g picric acid 25 g NaOH 100 mL water	Use a certified and tested hood. Immerse in boiling solution for 5 min. Precaution—Do not boil dry—anhydrous picric acid is unstable and highly explosive. Alternative: Electrolytic at 6 V for 40 s (room temperature). Use stainless steel cathode.
86	3 g oxalic acid 4 mL H ₂ O ₂ (30 %) 100 mL water	Use a certified and tested hood. Solution should be freshly prepared. Immerse 15–25 min when specimens or parts cannot be given usual metallographic polish. Multiple etching may be required.
87	10 mL HNO ₃ 20–50 mL HCl 30 mL glycerol	Use a certified and tested hood—Can give off nitrogen dioxide gas. Precaution—Mix HCl and glycerol thoroughly before adding HNO ₃ . Do not store. Properly discard before solution attains a dark orange color. Immerse or swab few seconds to few minutes. Higher percentage of HCl minimizes pitting. A hot water rinse just prior to etching may be used to activate the reaction. Sometimes a few passes on the final polishing wheel is also necessary to remove a passive surface.
88	10 mL HNO ₃ 20 mL HCl 30 mL water	Use a certified and tested hood—Can give off nitrogen dioxide gas. Precaution—Properly discard before solution attains a dark orange color. Immerse few seconds to a minute. Much stronger reaction than No. 87.



TABLE 2 Continued

Etchant	Composition	Procedure
89	10 mL HNO ₃ 10 mL acetic acid 15 mL HCl 2–5 drops glycerol	Use a certified and tested hood. Do not store. Immerse or swab few seconds to few minutes.
90	10 mL HNO ₃ 20 mL HF 20–40 mL glycerol	Use a certified and tested hood—Immerse 2–10 s. Do not store. Properly discard after use. Solution decomposes on standing.
91	5 mL HNO ₃ 5 mL HCl 1 g picric acid 200 mL ethanol (95 %) or methanol (95 %)	This etchant is equivalent to a 1 + 1 mixture of No. 80 and No. 74 (5 % HNO ₃). Swab for 30 s or longer.
92	10 mL HCl 100 mL ethanol (95 %) or methanol (95 %)	Immerse 5–30 min or electrolytic at 6 V for 3–5 s.
93	concentrated HNO ₃	Use a certified and tested hood. Electrolytic at 0.2 A/cm ² for few seconds.
94	2 g CuCl ₂ 40 mL HCl 40–80 mL ethanol (95 %) or methanol (95 %)	Submerged swabbing for few seconds to several minutes. Attacks ferrite more readily than austenite.
95	2 g CuCl ₂ 40 mL HCl 40–80 mL ethanol (95 %) or methanol (95 %) 40 mL water	Immerse or swab few seconds to few minutes.
96	85 g NaOH 50 mL water	Electrolytic at 6 V for 5–10 s.
97	45 g KOH 60 mL water	Composition of solution is approximately 10 N. Electrolytic at 2.5 V for few seconds. Stains sigma and chi yellow to red brown, ferrite gray to blue gray, carbides barely touched, austenite not touched.
98	10 g K ₃ Fe(CN) ₆ † 10 g KOH or NaOH 100 mL water	Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Use fresh. (a) Immerse or swab 15–60 s. Stains carbides and sigma. (To differentiate, No. 31 electrolytic at 4 V will attack sigma, but not carbides. If pitting occurs, reduce voltage.) (b) Immerse in fresh, hot solution 2–20 min. Stains carbides dark, ferrite yellow, sigma blue. Austenite turns brown on overetching. (c) Swab 5–60 s. (Immersion will produce a stain etch). Follow with water rinse, alcohol rinse, dry.
99	25 mL HCl 3 g ammonium bifluoride 125 mL water few grains potassium metabisulfite	Mix fresh. (For stock solution, mix first three items. Add potassium metabisulfite just before use.) Immerse few seconds to a few minutes.
100	10 g FeCl ₃ 90 mL water	Immerse few seconds.
101	2 g CrO ₃ 20 mL HCl 80 mL water	Use a certified and tested hood—Immerse 5–60 s. (CrO ₃ may be increased up to 20 g for difficult alloys. Staining and pitting increase as CrO ₃ increased.)
102	concentrated NH ₄ OH	Use a certified and tested hood. Electrolytic at 6 V for 30–60 s. Attacks carbides only.
103	20 mL HNO ₃ 4 mL HCl 20 mL methanol (99 %)	Use a certified and tested hood. Immerse 10–60 s.
104	5 mL HNO ₃ 45 mL HCl 50 mL water	Use a certified and tested hood. Immerse 10 min or longer.
105	5 mL H ₂ SO ₄ 3 mL HNO ₃ 90 mL HCl	Use a certified and tested hood. Precaution—add H ₂ SO ₄ slowly to HCl with stirring, cool; then add HNO ₃ . Properly discard when dark orange color. Swab 10–30 s.
106	7 mL HNO ₃ 25 mL HCl 10 mL methanol (99 %)	Use a certified and tested hood—Use fresh to avoid pitting. Immerse or swab 10–60 s.



TABLE 2 Continued

Etchant	Composition	Procedure
107	10 mL H ₃ PO ₄ 50 mL H ₂ SO ₄ 40 mL HNO ₃	Use a certified and tested hood. Precaution—Mix H ₃ PO ₄ and HNO ₃ thoroughly, then add H ₂ SO ₄ slowly with stirring. Use fresh, but allow to cool. Electrolytic at 6 V for few seconds. Brown discoloration will form at edges of specimen. To slow reaction, add water (to 100 mL) very carefully with stirring. Attacks bakelite mounts.
108	3–10 mL H ₂ SO ₄ 100 mL water	Electrolytic at 6 V for 5–10 s. Tends to pit with longer times.
109	50 mL HCl 25 mL HNO ₃ 1 g CuCl ₂ 150 mL water	Make fresh but allow to stand 30 min to avoid plating out copper. Immerse few seconds to a few minutes.
110	10 mL HCl 5 mL HNO ₃ 85 mL ethanol (95 %) or methanol (95 %)	Immerse to several minutes until deeply etched. Follow with light repolish.
111	5 mL H ₂ SO ₄ 8 g CrO ₃ 85 mL H ₃ PO ₄	Use a certified and tested hood. Electrolytic at 10 V (0.2 A/cm ²) for 5–30 s. Reveals Ti- and Cb-rich areas at a faster rate than grain boundaries.
112	60 mL acetic acid 30 mL H ₂ O ₂ (30 %)	Use a certified and tested hood. Immerse 8–15 s.
113	15 mL acetic acid 15 mL HNO ₃ 60 mL glycerol	Use a certified and tested hood. Do not store. Use fresh solution at 80°C (176°F).
114	15 mL acetic acid 20 mL HNO ₃ 80 mL water	Use a certified and tested hood. Use fresh solution at 40–42°C (104–108°F). Immerse 4–30 min depending on depth of worked metal layer. Clean with cotton in running water. Do not store.
115	100 mL acetic acid 10 mL H ₂ O ₂ (30 %)	Use a certified and tested hood. Immerse 10–30 min depending on depth of worked metal layer. Clean in HNO ₃ if necessary.
116	5–10 g AgNO ₃ 90 mL water	Swab.
117	10 mL HCl 90 mL water	(a) Immerse for ½–5 min. Follow with electrolytic etch at low current density in same solution. If specimen has considerable surface flow, immerse in concentrated HCl for a few seconds, then follow above procedure. (b) Immerse for ½–2 min.
118	1 mL HNO ₃ 75 mL diethylene glycol 25 mL water	Swab 3–5 s for F and T6, 1–2 min for T4 and O temper.
119	1 mL HNO ₃ 20 mL acetic acid 60 mL diethylene glycol 20 mL water	Use a certified and tested hood. Swab 1–3 s for F and T6, 10 s for T4 and O temper. Do not store.
120	10 mL HF 90 mL water	Immerse with gentle agitation 3–30 s.
121	0.7 mL H ₃ PO ₄ 4 g picric acid 100 mL ethanol (95 %) or methanol (95 %)	Composition critical. (a) Immerse with gentle agitation 10–30 s. (b) To increase staining immerse and withdraw with a meniscus layer. Lightly apply etchant over surface until dark stain develops.
122	2 g oxalic acid 100 mL water	Swab.
123	60 mL H ₃ PO ₄ 100 mL ethanol (95 %)	Electrolytic: Use stainless steel cathode. Space electrodes 2 cm apart. Start at 3 V dc. After 30 s maintain at 1½ V.
124	5 mL acetic acid 10 mL water 6 g picric acid 100 mL ethanol (95 %) or methanol (95 %)	Use a certified and tested hood. Immerse with gentle agitation 10–60 s.
125	10 mL acetic acid 6 g picric acid 100 mL ethanol (95 %) or methanol (95 %)	Use a certified and tested hood. Immerse with gentle agitation 15–30 s.
126	30 mL acetic acid 15 mL water 6 g picric acid 100 mL ethanol (95 %) or methanol (95 %)	Use a certified and tested hood. Immerse with gentle agitation 1–30 s.



TABLE 2 Continued

Etchant	Composition	Procedure
127	20 mL acetic acid 20 mL water 3 g picric acid 50 mL ethanol (95 %) or methanol (95 %)	Use a certified and tested hood. Immerse with gentle agitation 5–30 s.
128	8 mL HF 5 mL HNO ₃ 200 mL water	Use a certified and tested hood. Immerse with gentle agitation 5–15 s.
129	10 mL HF 30 mL HNO ₃ 60 mL lactic acid	Swab 10–20 s. Vary HF to increase or decrease activity.
130	25 mL HCl 75 mL methanol	Caution—Keep below 24°C (75°F). Electrolytic at 30 V for 30 s.
131	5 mL H ₂ SO ₄ 1 mL HF 100 mL methanol (95 %)	Use a certified and tested hood. Electrolytic at 50–60 V for 10–20 s.
132	5 mL HF 10 mL HNO ₃ 50 mL lactic acid	Use fresh. (a) Swab with heavy pressure for 5–10 s. Water rinse, alcohol rinse, dry, then etch with No. 98c. (b) Swab for 5–30 s.
133	50 mL HNO ₃ 50 mL acetic acid	Use a certified and tested hood. Do not store. <i>Mix fresh.</i> Immerse or swab 5 to 30 s. Will chemically polish with longer times. Sulfidized grain boundaries etched before normal grain boundaries. Do not store.
134	70 mL H ₃ PO ₄ 30 mL water	Electrolytic 5–10 V for 5–60 s. (Polishes at high currents.)
135	80 mL HNO ₃ 3 mL HF	Use a certified and tested hood. Warm specimen in boiling water prior to immersion for 10 to 120 s.
136	20 mL H ₃ PO ₄ 80 mL water	Electrolytic at 10–20 V for 10–15 s.
137	10 g NaNO ₃ 100 mL water	Electrolytic, 0.2 A/cm ² , 1 min.
138	5 g FeCl ₃ 2 mL HCl 100 mL ethanol (95 %) or methanol (95 %)	Swab 10–60 s.
139	5 g KCN 100 mL water 0.5 mL H ₂ O ₂ (3 %)	Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Immerse 10–100 s.
140	50 mL acetic acid 50 mL HNO ₃ 50 mL acetone	Use a certified and tested hood. Do not store. Decomposes with possible explosion on standing. Immerse 10–30 s.
141	3 g NH ₄ Cl 3 g CrO ₃ 10 mL HNO ₃ 90 mL water	Use a certified and tested hood—Swab 5–30 s. Do not store.
142	5 mL HF 10 mL glycerol 85 mL water	Electrolytic at 2–3 V for 2–10 s.
144	A 10 g sodium thiosulfate 100 mL water B 10 mL HCl 90 mL water	Electrolytic in Solution A: specimen is cathode, 10 V, 5–10 s. Then electrolytic in Solution B: specimen is anode, 10 V, 5–10 s.
145	2 mL H ₂ SO ₄ 100 mL water	Electrolytic at 3–10 V for 5–15 s. Use platinum wires. H ₂ SO ₄ may be increased to 20 mL for deeper attack.
146	10 mL HF 100 mL HNO ₃	Immerse 30 s–3 min.
147	20 mL HNO ₃ 80 mL HCl	Immerse 5–30 s.



TABLE 2 Continued

Etchant	Composition	Procedure
148	5 mL HNO ₃ 100 mL water	Immerse 10–30 s.
149	50 mL HCl 2 mL H ₂ O ₂ (30 %) 50 mL water	Immerse 10–30 s. Do not store.
150	60 mL HCl 20 mL HNO ₃ 40 mL glycerol	Use a certified and tested hood. Do not store. Swab few seconds to a minute. Properly discard when solution turns dark yellow.
151	10 mL HF 25 mL HNO ₃ 150 mL water	Swab 5–30 s.
152	85 mL NH ₄ OH 15 mL H ₂ O ₂ (30 %)	Use a certified and tested hood. Immerse 5–15 s. Do not store—Decomposes.
153	10 mL HNO ₃ 50 mL HCl 60 mL glycerol	Use a certified and tested hood. Do not store. Add HNO ₃ last. Properly discard when dark yellow. Immerse 10–60 s. Preheating specimen in boiling water hastens reaction.
154	50 mL HCl 50 mL ethanol (95 %) or methanol (95 %)	Immerse 10–100 s.
155	3 mL selenic acid 10 mL HCl 100 mL ethanol (95 %) or methanol (95 %)	Use a certified and tested hood. Immerse 1–15 min. (Up to 30 mL of HCl may be used for more vigorous action.) Stable for 3–90 days, depending on HCl concentrations. Use appropriate gloves.
156	1 g thiourea 1 mL H ₃ PO ₄ 1000 mL water	Electrolytic, 0.005–0.01 A/cm ² , 1–2 min.
157	25 g CrO ₃ 150 mL HCl 50 mL water	Use a certified and tested hood. Immerse 5–20 s.
158	10 mL HF 10 mL HNO ₃ 20 mL glycerol	Swab 5–15 s. Do not store Properly discard after use. Solution decomposes on standing.
159	5 mL HF 20 mL HNO ₃ 50 mL acetic acid	Use a certified and tested hood. Swab 10–30 s. Do not store.
160	20 mL HF 15 mL H ₂ SO ₄ 5 mL HNO ₃ 50 mL water	Immerse to 5 min.
161	25 mL HNO ₃ 5 mL HF	Immerse 5–120 s.
162	A 50 mL lactic acid 30 mL HNO ₃ 2 mL HF B 30 mL lactic acid 10 mL HNO ₃ 10 mL HF	Swab 1–3 min in Solution A (acts as etch polish). To etch, swab with Solution B for 5 s. Repeat if necessary. The HF may be varied to give more or less etching. Do not store.
163	30 mL H ₂ SO ₄ 30 mL HF 3–5 drops H ₂ O ₂ (30 %) 30 mL water	Immerse 5–60 s. Use this solution for alternate etch and polishing.
164	50 mL HNO ₃ 30 g ammonium bifluoride 20 mL water	Use a certified and tested hood. Swab 3–10 s.
165	10 mL HCl 90 mL ethanol	(a) Electrolytic at 10 V for 30 s. Use carbon cathode and platinum wire connection to specimen. For etch-polishing, use shorter time. (b) Electrolytic at 6 V for 10 s. Use stainless steel cathode and platinum or Nichrome wire contact to specimen.



TABLE 2 Continued

Etchant	Composition	Procedure
166	A 20 g ammonium persulfate 90 mL water B 20 g KCN 90 mL water	Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Mix 1 + 1 ratio of Solution A and B just before use. (A mixture of 5 drops of each will cover the surface of a 1 in. dia mount.) Immerse to several minutes.
167	5 g NaCN 100 mL water	Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Electrolytic at 1–5 V ac for 1–2 min. Use platinum cathode.
168	20 mL HCl 35 g NaCl 80 mL water	Composition given will saturate the solution with NaCl. Electrolytic at 1½ V ac for 1 min.
169	5 mL HNO₃ 50 mL ethylene glycol 20 mL ethanol (95 %) or methanol (95 %)	Electrolytic at 0.05 A/cm² for 2 min. Use stainless steel cathode.
170	1 mL HF 30 mL HNO₃ 30 mL lactic acid	(a) Swab 5–30 s. Follow with water rinse, alcohol rinse, dry. (b) Swab for 10 s intervals. Increase HF to exaggerate grain boundaries.
171	concentrated HCl	Use a certified and tested hood. Electrolytic at 5 V ac for 1–2 min. For etch-polishing, use shorter times. Follow with water rinse, alcohol rinse, and dry.
172	A 5 g ammonium persulfate 100 mL water B 5 g KCN 100 mL water	Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Prepare 1 + 1 mixture of Solutions A and B just before use. (A mixture of 5 drops of each will cover the surface of a 1 in. dia mount.) Immerse 1–2 min.
173	50 mL NH₄ OH 10–30 mL H₂ O₂ (50 %)	Immerse few seconds to a minute.
174	A 25 mL HNO₃ 1 g K₂ Cr₂ O₇ 1000 mL water B 40 g CrO₃ 3 g Na₂ SO₄ 200 mL water	Use a certified and tested hood. Prepare 1 + 1 mixture of Solutions A and B. Apply with camel's hair brush. Nonadherent film of silver chromate should form. If film adheres, add more of Solution A, if none forms, add Solution B.
175	1 g CrO₃ 1 mL H₂ SO₄ 1000 mL water	Use a certified and tested hood. Immerse to 1 min.
176	2 g FeCl₃ 100 mL water	Immerse 5–30 s.
177	10 g NaOH 100 mL water	Swab or immerse 5–15 s.
178	20 mL HF 20 mL HNO₃ 60 mL lactic acid	Swab for 5–20 s. Do not store.
179	A 10 mL HF 10 mL HNO₃ 30 mL lactic acid B 10 mL HF 90 mL H₂ SO₄	Use a certified and tested hood—Mix Solution B very slowly. Solution A is used as a chemical polish, though some etching will occur. Swab 2 or more minutes for desired surface. If surface is insufficiently etched use Solution B electrolytically at ½ –1 A/in.² of specimen. Use carbon cathode and platinum wire connection to specimen. Properly discard Solution B after 1 hr. Do not store.



TABLE 2 Continued

Etchant	Composition	Procedure
180	10 mL HNO ₃ 30 mL acetic acid 50 mL glycerol	Use a certified and tested hood. Immerse for ½ –10 min at 38 to 42°C (100–108°F). Do not store Properly discard after use. Solution composes on standing.
181	2 mL HCl 100 mL ethanol (95 %) or methanol (95 %)	Swab for 1–3 min.
182	10 mL HNO ₃ 10 mL acetic acid 80 mL glycerol	Use a certified and tested hood. Immerse for ½ –10 min at 38 to 42°C (100–108°F). Do not store. Properly discard after use. Solution decomposes on standing.
183	2 drops HF 1 drop HNO ₃ 25 mL glycerol	Immerse for 1 min. Do not store. Properly discard after use. Solution decomposes on standing.
184	10 g FeCl ₃ 2 mL HCl 100 mL water	Immerse for ½ –5 min.
185	10 mL HF 10 mL HNO ₃	Swab for few seconds.
186	10 mL HF 5 mL HNO ₃ 85 mL water	Swab 3–20 s.
187	10 mL HF 30 mL HNO ₃ 50 mL water	Swab 3–20 s.
188	1 mL HF 2 mL HNO ₃ 50 mL H ₂ O ₂ (30 %) 50 mL water	Use a certified and tested hood. Swab until stain is removed.
189	10 mL HF 25 mL HNO ₃ 45 mL glycerol 20 mL water	Swab 3–20 s. Do not store Properly discard after use. Solution decomposes on standing.
190	8 g KOH 10 mL H ₂ O ₂ (30 %) 60 mL water	Swab 3–20 s.
191	25 mL HF 18 g benzalkonium chloride 35 mL methanol (95 %) 40 mL glycerol	Swab 3–20 s.
192	1–3 mL HF 2–6 mL HNO ₃ 100 mL water	Swab 3–10 s or immerse 10–30 s. (HF attacks and HNO ₃ brightens the surface of titanium. Make concentration changes on this basis.)
193	2 drops HF 1 drop HNO ₃ 3 mL HCl 25 mL glycerol	Swab 3–20 s. Do not store Properly discard after use. Solution decomposes on standing.
194	20 mL HF 20 mL HNO ₃ 60 mL glycerol	Immerse 5–30 s. Do not store Properly discard after use. Solution decomposes on standing.
195	30 mL H ₃ PO ₄ 30 mL ethylene glycol 50 mL ethanol (95 %)	Electrolytic at 18–20 V (0.03 A/cm ²) for 5–15 min.
196	18 g CrO ₃ 75 mL acetic acid 20 mL water	Use a certified and tested hood. Dissolve CrO ₃ in hot water and cool before adding acetic acid. Keep solution below 2°C (35°F) during use. Electrolytic at 80 V for 5–30 min. Do not store.
197	5 g oxalic acid 100 mL water	Electrolytic at 6 V for 5–20 s.



TABLE 2 Continued

Etchant	Composition	Procedure
198	30 mL HF 30 mL HNO ₃ 30 mL glycerol	Swab for 60 s. Do not store. Properly discard after use. Solution decomposes on standing.
199	2 mL HF 5 g AgNO ₃ 100 mL water	Swab for 5 s.
200	A 40 g CrO ₃ 3 g Na ₂ SO ₄ 200 mL water B 40 g CrO ₃ 200 mL water	Use a certified and tested hood. Immerse in Solution A with gentle agitation for several seconds. Rinse in Solution B.
201	A 40 g CrO ₃ 1.5 g Na ₂ SO ₄ 200 mL water B 40 g CrO ₃ 200 mL water	Use a certified and tested hood. Immerse in Solution A with gentle agitation for several seconds. Rinse in Solution B.
202	A 10 g CrO ₃ 1 g Na ₂ SO ₄ 200 mL water B 40 g CrO ₃ 200 mL water	Use a certified and tested hood. Immerse in Solution A for 2–5 s. Rinse in Solution B.
203	20 g CrO ₃ 100 mL water	Use a certified and tested hood. Electrolytic at 0.2 A/cm ² for 5 s.
204	10 mL perchloric acid 10 mL glycerol 70 mL ethanol (95 %) 10 mL water	Use in a wash down/prechloric rated fume hood. Precaution—Keep cool when mixing and use. Electrolytic at 15–50 V for 15–60 s.
205	5 mL HF 2 mL AgNO ₃ (5 %) 100 mL water	Swab vigorously for 10–60 s. Wet cotton frequently.
206	5 mL HF 10 mL HNO ₃ 100 mL glycerol	Precaution—Properly discard after use. Solution decomposes on standing. Electrolytic at 9–12 V for 1–10 min.
207	30 mL HNO ₃ 30 mL acetic acid 30 mL water	Swab for 5–30 s. Do not store.
208	1 mL NH ₄ OH 3 g ammonium persulfate 100 mL water	Immerse or swab few seconds to a minute.
209	15 mL HNO ₃ 3 mL HF 80 mL water	Immerse 5–60 s.
210	50 mL water (cold) saturated with sodium thiosulfate 1 g potassium metabisulfite	First ingredient in stock solution. Add potassium metabisulfite before use. Solution good for several days, or longer. Immerse face up, gently agitate until coloration begins, allow to settle. Stop etch when surface is red-violet. Etch time varies with material. Colors matrix phases.
211	3 g potassium metabisulfite 10 g sodium thiosulfate 100 mL water	Use fresh solution. Immerse specimen face up, gently agitate solution until coloration begins, allow to settle. Stop etch when surface is red-violet. Etch time varies with material. Colors matrix phases.



TABLE 2 Continued

Etchant	Composition	Procedure
212	10–50 % HCl in water 0.5–1.0 g potassium metabisulfite per 100 mL of aqueous HCl solution Optional: 1 g CuCl ₂ 1–3 g FeCl ₃ 2–10 g ammonium bifluoride	For more corrosion resistant alloys. Increase the HCl and potassium metabisulfite contents. Use optional ingredients to improve coloration, if needed. Colors matrix phases. Use by immersion only.
213	2–10 mL HCl 0.5–3 mL selenic acid 100 mL ethyl alcohol (95 %)	For more corrosion resistant alloys, increase the HCl and selenic acid content. For highly corrosion-resistant alloys, use 20–30 mL HCl. Colors second phase constituents. Use by immersion only.
214	1 g sodium molybdate 100 mL water	Add nitric acid to lower the pH to 2.5–3. Add 0.1–0.5 g ammonium bifluoride for carbon steels. Use by immersion only. Colors carbides. Immerse about 15 s.
215	240 g sodium thiosulfate 30 g citric acid 24 g lead acetate 1000 mL water	Mix in order given. Store in a dark bottle at least 24 h before use at 20°C. Lightly pre-etch specimen before use. Use small portion of stock solution for 4 h max. Pre-etch steel specimens with nital before tinting the MnS (add 0.2 g sodium nitrite to 100 mL of etch) white. Colors phosphides in cast iron. Colors matrix of Cu alloys.
216	8–15 g sodium metabisulfite 100 mL water	Do not store. Mix fresh. Immerse specimen face up. Agitate solution gently until coloration begins, allow to settle. Stop when surface is dark. Use crossed polarized light and sensitive tint to improve coloration.
217	5 g ammonium bifluoride 100 mL water	Mix fresh, use plastic coated tongs and polyethylene beaker. Immerse until surface is colored.
218	3 g ammonium bifluoride 4 mL HCl 100 mL water	Mix fresh, use plastic coated tongs and polyethylene beaker. Immerse until surface is colored. Works best with attack-polished specimens.
219	60 mL HNO ₃ 40 mL water	Electrolytic etch, does not reveal twins in γ stainless steel. Excellent grain boundary etch for ferritic stainless steels. Use at 1 V dc, 120 s, with stainless cathode; 0.6 V dc with platinum cathode.
220	20 g NaOH 100 mL water	Electrolytic etch, colors δ-ferrite in stainless steels. Use at 2–20 V dc, 5–20 s, stainless steel cathode. If δ is not colored, increase NaOH to 40 g.
221	50 mL water 50 mL ethyl alcohol 50 mL methyl alcohol 50 mL HCl 1 g CuCl ₂ 2.5 g FeCl ₃ 2.5 mL HNO ₃	Use by immersion. Will not attack sulfides in stainless steels.
222	8 g Na ₂ SO ₄ 100 mL water	(a) Few seconds to 1 minute. (b) Pre-etch 2 s in No. 74, rinse, and etch 20 s.
223	A 8 g oxalic acid 5 mL H ₂ SO ₄ 100 mL water B H ₂ O ₂ (30 %)	Mix equal volumes of Solutions A and B just before use. Etch 2–3 s; 3 s pre-etch in No. 74 may be needed.
224	10 mL H ₂ O ₂ (30 %) 20 mL 10 % aqueous NaOH	Immerse 10 s at 20°C (68°F).
225	4 g NaOH 100 mL saturated aqueous KMnO ₄	Immerse 10 s at 20°C (68°F).
226	15 mL HCl 10 mL acetic acid 5 mL HNO ₃ 2 drops glycerol	Use a certified and tested hood. —Can give off nitrogen dioxide gas. Precaution —Mix HCl and glycerol thoroughly before adding HNO ₃ . Do not store. Properly discard before solution attains a dark orange color. Use fresh or age up to 1 min. Immerse or swab few seconds to few minutes. Can increase HNO ₃ to increase strength. Sometimes a few passes on the final polishing wheel is also necessary to remove a passive surface.

[†] Editorially corrected in May 2011.



TABLE 3 Etchant Names

Common Name	No.	Common Name	No.
Acetic glyceresia	89, 226	Groesbeck's	19
Alkaline Sodium Picrate	85	Hatch	2
Aqua regia	12	Howarth's	84
Barker's	5	Kalling's 1	95
Beraha's	99, 155, 211–215	Kalling's 2	94
Carapella	138	Keller's	3
Chrome regia	101	Klemm's	210
Contrast	141	Kroll's	192, 187
CP 4	60	Marble's	25
El-1R	107	Marshall's	223
Flat	133	Murakami's	98
Flouregia	90, 158	Nital	74
Frank's	104	Palmerton	200
Fry's	79	Phoschromic	111
G	107	Picral	76
Glyceresia	87	Ralph's	221
Gorsuch	75	Super Picral	77
Grard's No. 1	35	Vilella's	80
Green contrast	94	92-5-3	105

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